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2,2′-Dipropylmalonic acid: intermolecular association *via* strong O—H···O hydrogen bonds

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.051
wR factor = 0.150
Data-to-parameter ratio = 17.9

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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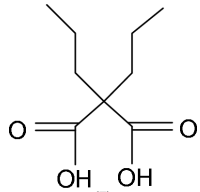
The molecular structure of the title compound, $C_9H_{16}O_4$, is described by a *trans*-planar configuration of the propyl chains. The molecule has an approximate pseudo-twofold rotation axis in the crystal structure. The molecules are interconnected by strong O—H···O hydrogen bonds and form an $R_2^2(8)$ ring structure.

Comment

The molecular assembly in the title compound, (I), is predominantly governed by N—H···O, O—H···O and O—H···N hydrogen bonds (Desiraju, 2002). Quite often, hydrogen-bonded patterns are formed which are interesting in supramolecular design and crystal engineering. Such patterns of hydrogen bonds were first analysed by Etter using graph theory (Etter, 1990) and later the formalism was further developed by Bernstein *et al.* (1995). In this report, we discuss 2,2′-dipropylmalonic acid, which is an intermediate for the preparation of the anticonvulsant drug valproic acid (Abbott & Acheampong, 1988). The intermolecular linkage *via* O—H···O hydrogen bonds forms a ring structure.



(I)

The molecular structure of (I) is shown in Fig. 1. The molecular conformation is characterized by four intersecting planes formed by propyl chains and carboxylic acid groups, and the molecule adopts a sort of propeller-shape. The maximum out-of-plane deviations from the least-square planes formed by the non-H atoms in the propyl chains and carboxylic acid groups are within 0.03 Å. The conformation of (I) is described by the torsion angles C1—C2—C4—C5 = −62.1 (2)°, C3—C2—C7—C8 = −56.9 (2)°, C2—C4—C5—C6 = 179.0 (2)° and C2—C7—C8—C9 = −177.9 (2)°. The last two are *trans*, indicating fully extended propyl chains. Compound (I) possesses molecular C_2 point group symmetry. However, the molecular symmetry is only approximately retained in the crystal structure. The r.m.s. deviation between related components is 0.06 Å.

The hydrogen-bond parameters are given in Table 1 and a packing diagram is shown in Fig. 2. The molecules are interconnected by strong O—H···O hydrogen bonds. Intermolecular interactions *via* O—H···O bonds form an $R_2^2(8)$ ring structure (Bernstein *et al.*, 1995).

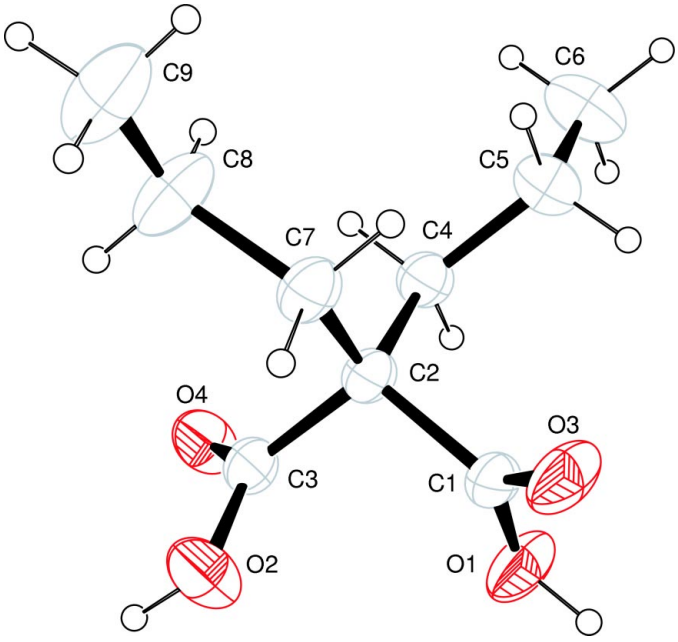


Figure 1
The molecular structure of (I). Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small circles of arbitrary radii.

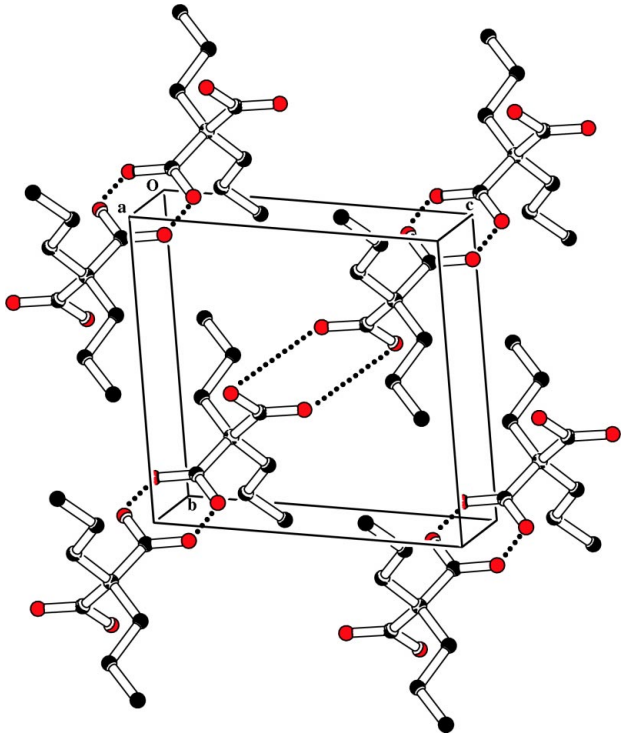


Figure 2
Packing diagram of (I), illustrating the intermolecular associations *via* strong O—H···O hydrogen bonds (dashed lines), forming a hydrogen-bonded $R_2^2(8)$ pattern. H atoms have been omitted.

Experimental

The title compound was synthesized by treating diethyl dipropylmalonate with sodium hydroxide at 353–363 K for 6 h. The melting point of (I) was 432 K. The compound was crystallized by slow evaporation of a saturated chloroform solution.

Crystal data

$C_9H_{16}O_4$
M_r = 188.22
Triclinic, $P\overline{1}$
a = 7.683 (1) Å
b = 8.549 (1) Å
c = 9.382 (1) Å
α = 76.249 (2)°
β = 70.140 (2)°
γ = 72.355 (2)°
V = 546.05 (12) Å^3
Z = 2
D_x = 1.145 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3369 reflections
θ = 5–55°
μ = 0.09 mm^{-1}
T = 293 (2) K
Block, colourless
0.55 × 0.22 × 0.12 mm

Data collection

Bruker SMART CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.977, T_{max} = 0.989
5840 measured reflections
2261 independent reflections
1784 reflections with $I > 2\sigma(I)$
R_{int} = 0.014
θ_{max} = 27.4°
$h = -9 \rightarrow 9$
$k = -10 \rightarrow 11$
$l = -12 \rightarrow 12$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.150
S = 1.04
2261 reflections
126 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0805P)^2 + 0.0901P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}

Table 1
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O3^{i}	0.83 (3)	1.85 (3)	2.678 (2)	176 (3)
O2—H2···O4^{ii}	0.87 (3)	1.80 (3)	2.673 (2)	175 (3)

Symmetry codes: (i) $-x, -y, 2-z$; (ii) $-x, 1-y, 1-z$.

The methyl and methylene H atoms were refined with fixed geometry (C—H = 0.96 and 0.97 Å), riding on their carrier atoms, with U_{iso} values set at 1.2 (1.5 for the methyl H atoms) times U_{eq} of the parent atom. The carboxy H atoms were located in a difference electron-density map and were refined isotropically.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT-Plus* (Bruker, 2001); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *PLUTON* (Spek, 1999); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PARST* (Nardelli, 1995).

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